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Potassium Perchlorate for Industrial Use

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Potassium Perchlorate for Industrial Use

Warning: According to the provisions of Clause 6 in GB 12268-2012, this product belongs to the oxidizing substances of item 5.1 in category 5. Care shall be taken during operation. Personnel using this standard shall have practical experience working in formal laboratory. This standard does not point out all possible safety issues. It is the user's responsibility to take appropriate safety and health measures and to ensure compliance with the conditions stipulated by relevant national regulations.

1 Scope

This Standard specifies the requirements, test methods, inspection rules, marking, labels, packaging, transportation, and storage of potassium perchlorate for industrial use.

This Standard is applicable to potassium perchlorate for industrial. This product is mainly used to manufacture fireworks and firecrackers, safety matches, meteorological rockets, civil ignition coal, oxidants, and automobile airbags, etc.

2 Normative References

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this document.

GB 190-2009 Packing Symbol of Dangerous Goods

GB/T 191-2008 Packaging - Pictorial Marking for Handling of Goods

GB/T 3051-2000 Inorganic Chemical Products for Industrial Use - General Method for Determination of Chloride Content - Mercurimetric Method

GB/T 6003.1-2012 Test Sieves - Technical Requirements and Testing - Part 1: Test Sieves of Metal Wire Cloth

GB/T 6678 General Principles for Sampling Chemical Products

GB/T 6682-2008 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 8170 Rules of Rounding off for Numerical Values & Expression and Judgement of Limiting Values

GB 10631-2013 Safety and Quality for Fireworks

GB 12268-2012 List of Dangerous Goods

GB 12463-2009 General Specifications for Transport Packages of Dangerous Goods

HG/T 3696.1 Inorganic Chemicals for Industrial Use - Preparations of Standard and Reagent Solutions for Chemical Analysis - Part 1: Preparations of Standard Volumetric Solutions

HG/T 3696.2 Inorganic Chemicals for Industrial Use - Preparations of Standard and Reagent Solutions for Chemical Analysis - Part 2: Preparations of Standard Solutions for Impurity

HG/T 3696.3 Inorganic Chemicals for Industrial Use - Preparations of Standard and Reagent Solutions for Chemical Analysis - Part 3: Preparation of Reagent Solutions

JJG 119-2005 Verification Regulation of Laboratory pH Meters

Provisions on the Administration of the Road Transport of Dangerous Goods

Rule for the Waterway Transport of Dangerous Goods

Provisions on Supervision and Administration of Transport Safety of Railway Dangerous Goods

Interim Provisions on the Administration of Transport of Railway Dangerous Goods

3 Molecular Formula and Relative Molecular Mass

Molecular formula: KClO_4

Relative molecular mass: 138.55 (according to 2016 international relative atomic mass)

4 Classification

Potassium perchlorate for Industrial use is divided into Type-I and Type-II; and its main uses are as follows:

---Type-I products are mainly used for meteorological rocket propulsion, oxidants,

6.3.4.1 Preparation of test solution

Take about 1g pre-ground powder sample, accurate to 0.0002g. Place it in a nickel crucible; add 6g of sodium nitrite ground into powder; and mix thoroughly. Cover it; put in a high-temperature furnace; gradually raise the temperature to 500°C; melt at this temperature for 1.5 h. Cool off; dissolve the melt by hot water and wash into the beaker. Cool off and transfer all to a 250 mL (V_1) volumetric flask; dilute to the mark by water; and shake well.

6.3.4.2 Test

Pipette 50 mL (V_2) of test solution into a 500mL conical flask; add 50mL of silver nitrate standard titration solution and 15mL of nitric acid solution by a pipette; and heat to all nitrogen dioxide gas escape. Cool off, add 5mL of nitrobenzene; and shake for 30s. Add 5mL of ferric ammonium sulfate indicator solution and use potassium thiocyanate standard titration solution to titrate until the solution appears red. Do a blank test at the same time.

6.3.5 Test data processing

The content of potassium perchlorate is calculated by the mass fraction w_1 of potassium perchlorate (KClO_4), and is calculated according to Formula (1):

$$w_1 = \frac{(V_0 - V)cM \times 10^{-3}}{m(V_2/V_1)} \times 100\% - (1.858 w_3 + 1.131 w_4) \quad \dots\dots (1)$$

Where:

V_0 - the volume of potassium thiocyanate standard titration solution consumed by titration of blank solution, in mL;

V - the volume of potassium thiocyanate standard titration solution consumed by titration of test solution, in mL;

c - Accurate value of the concentration of potassium thiocyanate standard titration solution, in mol/L;

M - The value of the molar mass of potassium perchlorate (KClO_4), in (g/mol) ($M=138.6$);

m - value of mass of the sample, in g;

V_1 - value of volume of test solution in 6.3.4.1, in mL;

V_2 - value of volume of the transferred test solution in 6.3.4.2, in mL;

6.5 Determination of chloride content

6.5.1 Principle

According to Clause 3 of GB/T 3051-2000.

6.5.2 Reagents and materials

According to Clause 4 of GB/T 3051-2000.

6.5.3 Test procedures

Take about 10g of sample, accurate to 0.01g. Place it in a 250mL conical flask; add 100mL of water; and heat until the sample is completely dissolved. Cool off, add 2~3 drops of bromophenol blue indicator solution; titrate the nitric acid solution (1mol/L) until it is just yellow, and then add 2~3 drops in excess. Add 1mL of phenylazoformic acid 2-phenylhydrazide indicator solution; and titrate by $c \left[\frac{1}{2} \text{Hg}(\text{NO}_3)_2 \right] \approx 0.05 \text{ mol/L}$ mercury nitrate standard titration solution until the solution turns from yellow to purple. Do a blank test at the same time. The treatment of mercury-containing waste liquid shall be carried out in accordance with Appendix D in GB/T 3051-2000.

6.5.4 Test data processing

The chloride content is calculated by the mass fraction w_3 of potassium chloride (KCl), and is calculated according to Formula (3):

$$w_3 = \frac{(V - V_0) c M \times 10^{-3}}{m} \times 100 \% \quad \dots\dots\dots (3)$$

Where:

V – value of volume of mercury nitrate standard titration solution consumed by titration of test solution, in mL;

V_0 – value of volume of mercury nitrate standard titration solution consumed by titration of blank solution, in mL;

c – accurate value of the concentration of mercury nitrate standard titration solution, in mol/L;

M – value of mole mass of chloride (by KCl), in (g/mol) ($M=74.55$);

m – value of mass of the sample, in g.

The arithmetic mean of the parallel determination results is taken as the determination

0.2mL, the adding amount of ferrous ammonium sulfate standard titration solution shall be appropriately increased (accurately add by a pipette). Do a blank test at the same time.

6.6.5 Test data processing

The content of chlorate is calculated by the mass fraction w_4 of potassium chlorate (KClO_3), and is calculated according to Formula (4):

$$w_4 = \frac{(V_0 - V)cM \times 10^{-3}}{m} \times 100 \% \quad \dots\dots\dots (4)$$

Where:

V_0 – value of volume of the potassium permanganate standard titration solution consumed by titration of blank solution, in mL;

V - value of volume of the potassium permanganate standard titration solution consumed by titration of test solution, in mL;

c – accurate value of the concentration of the potassium permanganate standard titration solution, in mol/L;

M – value of mole mass of the potassium chlorate ($\frac{1}{6}\text{KClO}_3$), in (g/mol) ($M=20.42$);

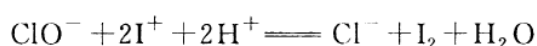
m – value of mass of the sample, in g.

The arithmetic mean of the parallel determination results is taken as the determination result; and the absolute difference between the two parallel determination results shall be no more than 0.003%.

6.7 Determination of hypochlorite content

6.7.1 Principle

The hypochlorite (ClO^-) in the test solution may oxidize the iodide ion into iodine (I_2); and use starch-potassium iodide test paper to determine whether there is hypochlorite in the sample. The main reaction formula is as follows:



6.7.2 Reagents and materials

Starch-potassium iodide test paper.

6.7.3 Test procedures

$$w_5 = \frac{(V - V_0)cM \times 10^{-3}}{m} \times 100 \% \quad \dots\dots\dots (5)$$

Where:

V – value of volume of the sodium thiosulfate standard titration solution consumed by titration of test solution, in mL;

V_0 – value of volume of the sodium thiosulfate standard titration solution consumed by titration of blank solution, in mL;

c – accurate value of concentration of the sodium thiosulfate standard titration solution, in mol/L;

M – value of mole mass of potassium bromate ($\frac{1}{6}\text{KBrO}_3$), in (g/mol) ($M=27.83$);

m – value of mass of the sample, in g.

The arithmetic mean of the parallel determination results is taken as the determination result; and the absolute difference between the two parallel determination results shall be no more than 0.002%.

6.9 Determination of sodium content

6.9.1 Principle

After the sample is dissolved in water, the sample is introduced into the flame; and the sample is evaporated, dissociated, atomized, and excited to emit light by the thermal and chemical effects of the flame (1800°C to 2500°C). According to the relationship between the emission intensity i of the characteristic spectral line and the concentration c of the measured element in the sample, that is, $i = acb$ (a and b are constants), determine the sodium content in the sample.

6.9.2 Reagents or materials

6.9.2.1 Potassium standard solution: 1mL solution contains 1mg of potassium (K).

6.9.2.2 Sodium standard solution: 1mL solution contains 1mg of sodium (Na).

6.9.3 Apparatus

Flame spectrophotometer.

6.9.4 Test procedures

6.9.4.1 Drawing of working curve

6.10.1.3 Apparatus

Micro burette: division of 0.02mL.

6.10.1.4 Test procedures

Take about 5g of sample, accurate to 0.0002g. Place it in a 250mL beaker; add 100mL of water; and heat to dissolve. Add 10mL of hydrochloric acid solution; and then make it slightly alkaline with ammonia water. Boil the solution and filter if necessary. Adjust to acidity with oxalic acid solution; then add 10 mL of excess oxalic acid solution; and heat to boil. Add 10 mL of ammonium oxalate solution under rapid stirring; and keep the pH value of the solution at 3.5~4.5 (check with accurate pH test paper). Keep warm on boiling water bath for more than 1h. Cool to room temperature and filter by quantitative filter paper. Wash the precipitate and filter paper by water and filter until there is no oxalic acid (tested by resorcinol test method). Collect the washing solution and filtrate and mix them together. This solution is Test Solution A and is used for the determination of magnesium salt content. Dissolve the precipitate by 10 mL of sulfuric acid solution (see 6.10.1.2.6) and thoroughly wash the filter paper by hot water. Combine the filtrate and the washing solution in a 250mL conical flask; dilute with water to about 100mL; heat to about 60°C; use potassium permanganate standard titration solution to titrate the solution until the solution appears pink and does not fade within 15s.

NOTE: The resorcinol test method -- take 1mL of the filtrate; put it in a test tube; add 2~3 drops of sulfuric acid solution (see 6.10.1.2.7) and a small amount of magnesium powder. When the magnesium powder is completely dissolved, add about 0.1g of resorcinol and shake until dissolved. After cooling, carefully add 3~4 drops of sulfuric acid along the tube wall. A blue ring is formed at the interface of the two liquids. When the lower sulfuric acid is heated, the blue color spreads downward from the interface, making the sulfuric acid layer blue.

6.10.1.5 Test data processing

The calcium salt content is calculated based on the mass fraction w_7 of calcium oxide (CaO), and shall be calculated according to Formula (7):

$$w_7 = \frac{V_c M \times 10^{-3}}{m} \times 100 \% \quad \dots\dots\dots (7)$$

Where:

V – value of volume of the potassium permanganate standard titration solution consumed by titration, in mL;

c – accurate value of the concentration of potassium permanganate standard titration solution, in mol/L;



6.12.2 Reagents or materials

6.12.2.1 Hydrochloric acid.

6.12.2.2 N-butanol.

6.12.2.3 Potassium thiocyanate solution: 30g/L.

6.12.2.4 Iron standard solution: 1mL solution contains 0.1mg of iron (Fe).

Pipette 10mL of iron standard solution prepared according to HG/T 3696.2 into a 100mL volumetric flask; dilute to the mark with water; and shake well. This solution is prepared before use.

6.12.3 Apparatus

Colorimetric tube: 50mL.

6.12.4 Test procedures

6.12.4.1 Preparation of test solution

Take $5\text{g} \pm 0.01\text{g}$ of sample; place it in a 100mL beaker; add about 25mL of water; and heat the solution on an electric stove. Take off; cool; add 5mL of hydrochloric acid; and transfer all to a colorimetric tube.

6.12.4.2 Test

Add 10mL of potassium thiocyanate solution and 10mL n-butanol to the test solution; shake for about 30s; and place when the layer appears; the color of the organic layer shall be no darker than the standard colorimetric solution.

The standard colorimetric solution is to take 1.00mL of iron standard solution; except that no sample is added; the other operations and the used reagents are the same as the test solution.

6.13 Determination of pH

6.13.1 Principle

According to the Nernst equation, the ion activity is proportional to the electrode potential; so, the relationship curve between the electrode potential and the activity maybe established for the measured solution; and the hydrogen ion concentration (pH) may be determined by measuring the potential.

m_1 - value of mass of sieve residue, in g.

The arithmetic mean of the parallel determination results is taken as the determination result; and the absolute difference between the two parallel determination results shall be no more than 0.2%.

6.15 Determination of looseness

6.15.1 Principle

Store the bagged samples in the stack for a certain period of time; freely fall from a certain height on a hard surface; and weigh the mass of the sample left on the sieve after sieving.

6.15.2 Apparatus

6.15.2.1 Test sieve: It is 950mm long and 600mm wide; and is equipped with a wooden frame about 120mm high; and the screen aperture is 4.75mm.

6.15.2.2 Stopwatch.

6.15.2.3 Platform scale: 10kg, with a division value of 0.1kg.

6.15.3 Test procedures

From the bagged products in the warehouse where the storage period in stack is no more than 3 months; select the seventh layer of bagged products from top to bottom for testing sample.

After weighing the test sample, use a mechanical or manual method to freely drop it from a height of 1m onto a flat, hard surface. Turn the bag over, then pour the sample in the bag onto the test sieve; and sieve at a frequency of 1s^{-1} . The sieving stroke is about 400mm, the sieving time is 1min; and a stopwatch is used to accurately time. After sieving, wight the mass of the sieve residue. The number of test bags shall be no less than 3 bags.

6.15.4 Test data processing

The looseness is calculated based on the mass fraction w_{12} of the sample with a particle size of less than 4.75mm, and shall be calculated according to Formula (12):

$$w_{12} = \frac{1}{n} \sum_{i=1}^n \left(\frac{m - m_i}{m} \right) \times 100 \% \quad \dots\dots\dots (12)$$

Where:

the entire batch of products shall be unqualified.

7.5 Use the rounding-off value comparison method stipulated in GB/T 8170 to determine whether the inspection result meets this Standard.

8 Markings and Labels

8.1 The packaging bags of potassium perchlorate for industrial use shall have a firm and clear mark, the contents of which include manufacturer name, address, product name, type, grade, net content, batch number or production date, this standard number, "oxidative substance" mark specified in Clause 3 of GB 190-2009, the "fear of sun" and "fear of rain" marks specified in Clause 2 of GB/T 191-2008.

8.2 Each batch of potassium perchlorate for industrial use that leaves the factory shall be accompanied by a quality certificate, the contents of which include name of the manufacturer, address, product name, type, grade, net content, batch number or production date, and this Standard number.

8.3 In addition to the quality certificate, the qualified products in each batch or in each sales unit of Type-II potassium perchlorate for industrial use that leave the factory shall also be accompanied by a product use reminder, the contents of which include "If this product is used to manufacture fireworks, firecrackers, the use amount of potassium perchlorate shall ensure that the chlorate in pyrotechnics complies with the provisions of 5.6.1.1 in GB 10631-2013" words, as well as the potassium perchlorate contents and chlorate contents in this batch of products.

9 Packaging, Transportation and Storage

9.1 The packaging type of the potassium perchlorate product for industrial use shall comply with the provisions of Table 1 in GB 12268-2012; and the limited mass of the packaging shall comply with the provisions of Appendix A in GB 12463-2009. When using rail transportation, its packaging shall also comply with the provisions of the *Interim Provisions on the Administration of Transport of Railway Dangerous Goods*. The inner packaging shall adopt polyethylene plastic bag. After the air in the bag is exhausted, the mouth of the bag is tightly tied. The packaging quality of potassium perchlorate products for industrial use shall satisfy the Class-II packaging performance test specified in GB 12463-2009. The net content of each piece is 25 kg or 50 kg. When users have special requirements for packaging specifications, it can be negotiated between the supplier and the purchaser. The packaging type shall comply with the provisions of Table 1 in GB12268-2012; and the limited quality of the packaging shall comply with the provisions of Appendix A in GB 12463-2009.

9.2 The transportation of potassium perchlorate for industrial use shall comply with the

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